California Environmental Protection Agency

Air Resources Board

SOP MLD 016

STANDARD OPERATING PROCEDURE FOR THE MASS ANALYSIS AND SUBSEQUENT EXTRACTION OF SSI-SAMPLED PM_{10} AND TSP FROM EXPOSED QUARTZ AND GLASS MICROFIBER FILTERS

Northern Laboratory Branch
Monitoring and Laboratory Division

First Approved Date of SOP: May 1, 1986
Approval Date of Last SOP Amendment: January 18, 2002
Revision Number: 5.0

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1.0 SCOPE

This document details the mass analysis and water extraction of particulate matter less than or equal to ten micrometers in diameter (PM_{10}) from exposed quartz microfiber filters and total suspended particulate (TSP) from glass microfiber filters. It combines and supercedes S.O.P NSL016 (Revision No. 0) (May 1, 1986 and March 21, 1991) and SOP MLD 030 (April 1, 1990).

2.0 SUMMARY OF METHOD

Individual quartz and glass microfiber filters are weighed on an electronic balance, which is interfaced to the Laboratory Information Management System (LIMS), before field sampling. Particulate matter less than or equal to ten micrometers in diameter is collected from ambient air over a 24-hour period on quartz filters. Total suspended particulate is collected from ambient air over a 24-hour period on glass filters. The filters are then post-weighed (LIMS calculates the particulate mass per cubic meter of air) and a 3 1/2" x 4 1/2" section of the exposed portion is cut from each filter. The PM_{10} or TSP deposited on this section is extracted by vigorous shaking in 100 mL of deionized water. The extraction solution is vacuum filtered, bottled and stored in a refrigerator for later analysis by ion chromatography.

3.0 INTERFERENCES

3.1 Since fingerprints increase the mass of the quartz filters and also contaminate them, gloves must be worn when handling quartz filters.

3.2 The moisture content of a filter will affect its weight; therefore, filters must be equilibrated in a conditioning environment for at least 24 hours before being weighed. During this equilibration period, the relative humidity must remain at 37% $\pm$ 5% and the temperature at 21.3 $\pm$ 3.0◦C (65-76°F). If the relative humidity and temperature fall outside of this range at any time during the equilibration period, the equilibration must be started over. The goal during the equilibration period will be to maintain a relative humidity of 35% ±5% and a temperature of 20-23°C (68-73.5°F).

The temperature and relative humidity are recorded on a Dickson

Humidity/Temperature weekly chart recorder. The humidity/temperature recorder is calibrated every three months using a NIST-traceable humidity/ temperature monitor. The humidity (%RH) and temperature (°C) must be within $\pm$2 of the NIST standard value.

3.3 Contamination of samples can occur from failure to clean the cutting board and scissors. All cutting equipment should be wiped thoroughly with a dry laboratory wipe prior to each use.

3.4 During sample preparation and extraction rinsing, use only deionized water that is monitored for resistivity, such as Nanopure. Nanopure water should have a minimum resistivity of 10 megaohms. Regular tap water or deionized tap water may cause contamination. Always rinse the end of the Nanopure delivery tube with Nanopure water prior to each use.

3.5 Unexposed filter pieces are used as the background media for method blanks and spikes; individual, unexposed filters should be quartered and the pieces placed together in a glassine enclosure. Careful storage of these pieces is essential for quality control. Place each enclosure in its own numbered folder for identification purposes if the blank is contaminated. Manila folder is used for PM_{10} filters and green folder for TSP filters.

3.6 All glassware and sample bottles, including lids, should be washed thoroughly and rinsed with Nanopure water prior to use. A dishwasher using non-foaming liquid detergent with a deionized tapwater rinse cycle can be used for the glassware and sample bottles, followed by rinsing with Nanopure quality water. Using excess dishwasher detergent will cause a spotty residue on the glassware and sample bottles. Detergent residue left on the washing machine door after cycling is a clear indication of excess detergent.

4.0 APPARATUS AND MATERIALS

4.1 Light box, 16" x 18"

4.2 Sartorius Analytic Balance interfaced to LIMS, minimum resolution of 0.1 mg and precision of 0.5 mg)

4.3 Filter weighing rack for Sartorius balance

4.4 Ainsworth 3 g and 1 g Class S calibration weights and plastic tweezer

4.5 Staticmaster anti-static devices

4.6 Computer terminal interfaced to LIMS

4.7 Dickson Model THDX Humidity/Temperature Recorder

4.8 Wheaton Unispense II water delivery system, minimum precision in the 100 ml range of 0.3 ml

4.9 Dispensing jug, 20 liter capacity

4.10 Erlenmeyer flasks, 250 mL, equipped with ground-glass stoppers

4.11 Sample bottles, 125mL, polypropylene or high-density polyethylene, equipped with screw-top lids

4.12 Cutting board with ruled edges

4.13 Scissors, long edge

4.14 Filter dome, 2000 mL

4.15 Filtration membranes, 47 mm in diameter, 0.45 μm pore diameter

4.16 Magnetic filter funnel, polysulfone, 300 mL capacity, for use with 47 mm filtration membranes

4.17 Shaker table with prongs fitted for 250 mL Erlenmeyer flasks

4.18 Dishwasher with deionized water rinse capability, industrial strength detergent

4.19 Refrigerator with multiple shelves

4.20 Rainin Micropipettor, 2.5 ml capacity

4.21 Miscellaneous: Manila folders (9" x 12", 5th cut); glassine enclosures (8 1/2"x 10 1/2"); 24-Hour Air Sample Report forms; disposable laboratory wipes (kimtowels); self-adhesive labels; waterproof ink pen; clock or timer; disposable, plastic gloves; bleach

5.0 FILTER INSPECTION AND PRE-WEIGHING PREPARATION PROCEDURE

5.1 Quartz (Whatman QMA, 8''x10'' and glass microfiber filters (Whatman EPM 2000, 8" x 10"), as received from the U.S.EPA, need to be inspected for manufacturing defects. Accomplish this by viewing both sides of a filter while it is on a light table.

5.2 Reasons for rejection include tears, pinholes, frayed edges, coloration, built-up clumps of fibers (visible on the light table as dark gray areas), thin areas and blue or black colored specks. (See Appendix C for detailed filter inspection procedures and criteria.)

5.3 Prepare filters for pre-weighing by placing them into manila folders, followed by 8 1/2" x 10 1/2" glassine enclosures, followed by 24-Hour Air Sample Report forms.

5.4 Allow the filters to equilibrate at least 24 hrs in the Balance Room prior to pre-weighing.

6.0 BALANCE CALIBRATION PROCEDURE

6.1 Calibrate the balance, both internally and externally, each day prior to filter weighing. Perform all calibrations and weighings with the glass enclosure door closed.

6.2 Clean the balance pan and the filter holder with a soft brush and check the bubble level (re-level if necessary).

6.3 Remove the filter holder, push the "**T**" button, wait for the display to read 0.0000 g. Then push the **CALIBRATE** switch, wait for the display to again read 0.0000 g.

6.4 Place the filter holder back onto the balance pan, push the **"T"** button, wait for the display to read 0.0000g. Then place the 1 and 3 g Class S weights onto the filter holder, wait until the display stabilizes, then record the weight, along with the temperature and relative humidity, in the Balance Log Book. The weight must be 4.0000±0.0005 grams. The temperature and relative humidity must be within the control limits.(See section 3.2)

6.5 If the weight falls outside of the range, repeat Section 6.3 through 6.4.

6.6 Open Procomm4 and select SQL*LIMS from the left drop-down box at the telnet prompt. Enter a username and password to log onto LIMS. Select LOG, then ENTER, and BY SAMPLE PLAN to begin entering data. Enter the appropriate balance ID, temperature, humidity, observed weight, and appropriate program (PM_{10} or TSP). Record the LIMS number in the Balance Log Book.

7.0 FILTER PRE-WEIGHING PROCEDURE

7.1 Before beginning the pre-weighing of the filters, check that the temperature and relative humidity of the Balance Room have remained, and are currently, within the allowable limits (See section 3.2) at least 24 hrs prior to the actual weighing. Be sure that the balance has been calibrated (See section 6.0) and the display reads 0.0000 g prior to each weighing (re-tare as necessary).

7.2 Open Procomm4 and select Sql*lims2 from the right drop-down box. Select yes to initiate the weighing session. Select the "PRE" option, then the appropriate program (PM_{10} or TSP).

7.3 Remove a filter from its folder (wearing gloves when handling quartz filters) and carefully place it into the filter holder on the balance pan. Be cautious edges of the filter are easily damaged.

7.4 Close the glass door and enter the factory stamped filter number at the LIMS prompt and press enter on the keyboard. While waiting for the balance to stabilize, record the filter number on the 24-Hour Air Sample Report form, then initial and date the form.

7.5 When the balance display has stabilized, push the **PRINT** button on the balance. Copy this displayed weight onto the 24-Hour Air Sample Report form.

7.6 Remove the filter from the balance and replace it along with its 24-Hour Air Sample Report form into its folder.

7.7 After making sure that the balance display reads 0.0000 g, repeat sections 7.3 through 7.6 for the remaining filters.

7.8 Every eighth filter is re-weighed as a quality control check. After the 8th filter has been weighed, the 1st filter is re-weighed; after the 16th filter is weighed, the 9th filter is re-weighed, etc. Write the replicate weight to the right of the first pre-weight on the 24-hour report form. The replicate difference limit for PM_{10} pre-weight is ±0.0028 grams. If the weight difference exceeds the quality control limit, all eight filters in the same batch must be re-weighed.

7.9 After pre-weighing is complete, a text file is generated that must be transferred into LIMS. This is accomplished through a file transfer protocol (FTP). Note the filename assigned to the file, then enter the

following at the MS_DOS prompt:

```
cd c:\temp\
ftp
open 146.114.112.200
(enter username)  balance
(enter password)  balance
put (filename)
quit
exit
```

8.0 FILTER LOG IN PROCEDURE

8.1 Filters received from the field must be inspected prior to equilibration in the Balance Room. While wearing gloves (for PM_{10} filters only), remove a filter from its protective glassine enclosure (the filter should have been folded in half by the field staff). Inspect the filter and Dickson flow rate record chart for any problems that may cause invalidation of the sample (see Appendix A). Place the filter back into its manila or green folder with the factory-stamped number facing out. Check to be sure that the factory-stamped number on the filter agrees with the filter number on the 24-Hour Air Sample Report form.

8.2 Place the Dickson chart on top of the glassine enclosure and the 24-Hour Air Sample Report form on top of the Dickson chart. Staple them and place them on top of the filter. Initial and record the receipt date on the 24-Hour Report Form. Label the folders with the site name, sample start date, mass only (*) or no mass (NM) (as applicable) and make-up run (as applicable). Orient the folders with the open edge up so that equilibration can occur more readily.

8.3 Organize the filters any convenient way (for example, alphabetically, or such that those needing extraction precede those needing mass-only analysis). Open Procomm4 and select SQL*LIMS from the left drop-down box at the telnet prompt. Enter a username and password to log on into LIMS. Select LOG, then ENTER, and BY SAMPLE PLAN to begin entering data. Enter the appropriate PM_{10} or TSP site name. Enter the relevant information obtained from the 24-Hour Air Sample Report form and the Dickson chart. Record the LIMS number PM_{10} or TSP site name. Enter the relevant information obtained from the 24-Hour Air Sample Report form and the Dickson chart. Record the LIMS number on the 24-Hour Air Sample Report form.

8.4 LIMS assigns replicate analyses to every tenth sample by counting the number of samples logged in for each test.

8.5 Record the barcode, and any invalidation or make-up run information in the PM_{10} Login Notebook in the Balance Room.

9.0 FILTER POST-WEIGHING PROCEDURE

9.1 After the filters have equilibrated at least 24 hrs under the required conditions of humidity and temperature (see Section 3.2), generate a mass worklist from the LIMS reports directory. Select the **pmwrkms.rep** file for PM_{10} post-weighing or the **tpwrkms.rep** file for TSP post-weighing The worklist will show which samples should be run as replicates. The filter designated as a replicate and the filters before the replicate on the worklist will be considered a batch.

9.2 Open Procomm4 and select Sql*lims2 from the drop-down box on the right. Select yes to initiate the weighing session. Select the "POST" option, then the appropriate program (PM_{10}).

9.3 Make certain that the balance has been calibrated (see Section 6.0) and that the display reads 0.0000 g. Remove the first replicate filter on the list from its folder (wearing gloves when handling the quartz filters) and place it into the filter holder on the balance pan.

9.4 Check to be sure that the factory-stamped number on the filter agrees with the filter number on the 24-Hour Air Sample Report form.

9.5 When the balance display has stabilized, push the **PRINT** button on the balance.

9.6 Copy this displayed weight onto the 24-Hour Air Sample Report form, then initial and date the form. Remove the filter from the balance and return it to its folder. If it is from a mass-only site place it inside the glassine enclosure in the folder.

9.7 After making certain that the balance display reads 0.0000 g, weigh the remaining filters from the same batch and repeat Sections 9.4 through 9.6.

9.8 Place the **replicate filter** into the filter holder again. Repeat Section 9.3 through 9.6, but recording the weight on the 24-Hour Air Sample Report form in the space above the first post-weight value. Place the filter in its folder. The replicate difference limit for PM_{10} post-weight is ±0.0050 g. If the weight difference exceeds the quality control limit, all the filters in the same batch must be re-weighed.

9.9 When post-weighing is complete, a text file is generated that must be

transferred into LIMS. This is accomplished through a file transfer protocol (FTP). Note the filename assigned to the file, then enter the following at the MS_DOS prompt:

```
cd c:\temp\
ftp
open 146.114.112.200
(enter username)  balance
(enter password)  balance
put (filename)
quit
exit
```

Once the file has been transferred into LIMS, a confirmation report will print. Generate a post-weight report by selecting **pmpost.rep** for PM_{10} samples or **tppost.rep** for TSP samples. Enter the filename printed at the top of the confirmation report. Once the post-weight report is generated, place it in the PM_{10} post-weight summary binder or TSP post-weight summary binder.

10.0 SAMPLE EXTRACTION PROCEDURE

10.1 Create and print an extraction worklist from LIMS reports using the file name **extprnt.rep**. Select the PM_{10} extraction method or the TSP sulfate extraction method. Take this copy into the Extraction Laboratory, along with the manila or green folders containing the filters.

10.2 Create a set of extraction labels with the sample number and barcode that correspond to the extraction worklist. Using a waterproof pen, clearly write the extraction date and the analyst's initials on each label. For those samples that are duplicated, append a **D** to the LIMS identification number.

Sets of method blanks and spikes equal to the number of duplicate samples on the extraction worklist are extracted along with the samples. The identification numbers of blanks and spikes (See section 3.5) are appended to the worklist. Randomly assign blank and spike ID numbers (1 – 10) for the extraction batch. On each label write the blank or spike I.D. number, the extraction date, and the analyst's initials.

10.3 For each sample, blank, and spike listed on the extraction worklist, place an Erlenmeyer flask on a moveable cart. Fold down one corner of each previously prepared labels and affix onto the Erlenmeyer flasks so that the labels are easily removable but do not fall off during the shaking procedure.

10.4 Thoroughly rinse the 20 liter dispensing jug with Nanopure water (see

Section 3.6). Fill the jug to the line (about 4 liters) with Nanopure water. Connect the jug to the Wheaton Unispense II water delivery system and deliver water to waste until all air bubbles have been cleared out of the line. (Tilting the jug will aid in the release of air bubbles trapped near the outlet.) Calibrate the system after filling the jug with Nanopure water by dispensing water into tared sample bottles, weighing the bottles and calculating the volume dispensed.

10.5 Using a dry, disposable kimtowel, thoroughly clean all of the cutting equipment. Place clean, open kimtowels around the immediate cutting area to prevent contamination.

10.6 Use the cutting board to cut a 3 1/2" x 4 1/2" section of the exposed area of each filter. Remove a filter from its manila or green folder (wearing gloves for the PM_{10} samples) and place it lengthwise on the cutting board, exposed-side up, with the factory-stamped number on the right side. Line the left margin of the exposed area up with the 4 1/2" line of the cutting board and cut the filter. (Note: If the exposed area is not centered on the filter, this cut will **not** produce two equal filter halves, but **will** produce two equal exposed-area halves.) Rotate the left half of the filter 90 degrees counter-clockwise, line the left margin of the exposed area up with the 3 1/2" line of the cutting board and cut the filter. These two cuts will produce a section of filter containing one quarter (3 1/2" x 4 1/2") of the original exposed area. The section removed should never be the one imprinted with the factory-stamped number. Retain the factory-stamped section for identification purposes. Place the remaining three-quarters of the filter back into the glassine enclosure.

10.7 Using the long-edge scissors, cut the 3 1/2" by 4 1/2" section into approximately equal-sized pieces. Cut the pieces directly into the Erlenmeyer flask labeled with the corresponding bar code number. (Blank and spike filters are pre-cut in quarters in manila and green folders (See section 3.5) next to the cutting board).

10.8 Dispense 100.0 ml of Nanopure water into the Erlenmeyer flask and seal with a ground glass stopper. For spikes, dispense 100.0ml of Nanopure water into an empty flask. Remove 2.0 ml of water from the flask using the micropipettor. Add 2.0ml of spike solution to the flask and then cut the filter into the flask.

10.9 Repeat Sections 10.6 through 10.8 for the remaining samples on the extraction worklist. For duplicate samples, use the section diagonally opposite the originally removed section.

10.10 Place the stoppered Erlenmeyer flasks securely on the shaker table

and shake them for 1.0 hr at low speed.

10.11 After the samples, blanks, and spikes have shaken for an hour, the extraction solutions are vacuum filtered in the order shown on the extraction worklist.

10.12 Thoroughly rinse the magnetic filter funnel with Nanopure water. With the vacuum on, insert the bottom piece of the magnetic filter funnel into the filter dome. Then add the filtration membrane and the top piece of the filter funnel. Pour about 75-100 ml of Nanopure water into the funnel to wet and rinse the surface of the membrane. Ensure that all the rinse water is removed by breaking the vacuum and re-applying it one or more times as necessary. Discard the rinse water to waste.

10.13 Immediately prior to filtering, transfer the label from the sample's Erlenmeyer flask directly to the individual sample bottle into which the filtrate will be stored. The label should adhere firmly and completely to the bottle. Place the labeled bottle underneath the filter funnel.

10.14 Swirl the contents of the flask and then pour the solution, along with filter pieces, into the filter funnel. Let the vacuum pull the liquid through the membrane into the sample bottle. Place a screen in the sink to collect filter pieces and prevent them from clogging the drain. Rinse the flask with water to remove any filter pieces remaining in the flask.

10.15 After the liquid has been pulled through, disconnect the apparatus carefully and seal the bottle with a clean lid.

10.16 Discard the used membrane and rinse both parts of the filter funnel thoroughly with Nanopure water in order to remove any filter residue or particles. Install a new membrane and wet it, and rinse the vacuum flask, as described in Section 10.12. Repeat Sections 10.12 through 10.15 for the remaining samples, blanks, and spikes on the extraction worklist.

10.17 The extraction solutions are now ready to be analyzed by ion chromatography. Place the filled sample bottles in shallow boxes. Store the blanks and spikes in a separate box. Store the boxes in chronological order in the refrigerator to prevent loss of volume and inhibit microbial growth.

10.18 Thoroughly rinse the filter funnel with Nanopure water and put the vacuum flask, along with the Erlenmeyer flasks and their stoppers, through two steam dishwasher cycles (once with soap added, then without soap).

10.19 When the samples are discarded after analysis, rinse the plastic sample bottles with tap water. Rinse the bottle caps in tap water and soak them in a 4-liter beaker filled with water and 50 ml of bleach. Check to ensure the drain stopper is securely seated. Place the sample bottles upright in the sink. Cover the bottles with metal racks and place an additional layer of bottles on top of the metal racks. Cover the second layer with metal racks and use the 4-liter beaker filled with sample bottle caps as a weight to prevent the bottles from tipping over. Soak the sample bottles overnight in a sink filled with Nanopure water and 300 ml of bleach (to kill any microbial growth). Rinse thoroughly with tap water, and then place in the dishwasher and wash twice without steam (once with soap added, then without soap). Rinse thoroughly with Nanopure water, dry, and reuse.

11.0 References

U.S. Environmental Protection Agency. 1997. Code of Federal Regulations, Title 40, Part 50, Appendix B. Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method)

U.S. Environmental Protection Agency. 1997. Code of Federal Regulations, Title 40, Part 50, Appendix M. Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere.

National Exposure Research Laboratory. 1997. Quality Assurance Guidance Document 2.11, Monitoring PM_{10} in Ambient Air Using a High-Volume sampler Method.

Appendix A: QUALITY CONTROL CRITERIA FOR PM_{10} and TSP FILTER SAMPLES

1.0 SCOPE

This document lists the quality control invalidation criteria for PM_{10} quartz filter samples collected on Size Selective Inlet (SSI) samplers and TSP glass filter samples. All samples collected in the field are to be checked using these criteria. If a sample does not meet these criteria, the sample is INVALID. The criteria listed supersede and replace any inspection criteria published prior to November 1996.

2.0 PM_{10} and TSP SAMPLE INVALIDATION CRITERIA

2.1 Filter Contamination

Filters that are dropped or become contaminated by any foreign matter (i.e. dirt, fingerprints, ink, liquids, etc.) that will affect the mass analysis or any ionic analysis of the filter are INVALID.

2.2 Damaged or Torn Filters

Filters with tears or pinholes, which occurred before or during sampling, are INVALID.

Filters missing any part of the exposed area are INVALID. Filters missing unexposed pieces of corners or edges are INVALID, unless the pieces are included in the returned sample package.

2.3 Filter Leakage

If the filter shows signs of air leakage due to a worn or improperly seated gasket, the sample is INVALID.

Gasket leakage is usually discernible as a dark streak along the edge of the filter, between the exposed area and the edge of the filter.

2.4 Dickson Recorder Chart

A complete Dickson recorder chart, documenting the flow rate through the sampler for 24 hours, must be submitted to the laboratory with each filter sample. Filter samples without complete Dickson recorder chart records are INVALID,

with the following exceptions:

2.4.1 Volumetric Flow Controlled Samplers (VFC)

In cases of inking problems where the trace is not complete for 24 hours, the sample will be considered VALID if the previous sample and subsequent sample are valid with complete chart traces. In instances when either the previous or subsequent sample is invalid, the operator must provide substantiating information for validating a sample in the Comments section of the 24-Hour Air Sample Report form. These samples will be considered for validation on a case-by-case basis.

2.4.2 Mass Flow Controlled Samplers (MFC)

In cases of inking problems where the trace is not complete, the sample will be considered VALID if: 1) start and stop marks are evident to verify start and stop times; 2) no more than three consecutive hours of trace are missing and the trace appears to be at the same value at either end of the skip; and 3) the operator validates that the sampler operated properly in the Comments section of the 24-Hour Air Sample Report Form. The flow rates from MFC samplers are generally not as stable as the VFC samplers, therefore the laboratory cannot confirm that the missing trace is the same value at either end of the skip.

2.5 Start/Stop Times

The sampler start and stop time must be 0000 (midnight) $\pm$ 30 minutes. If the Dickson recorder chart indicates that the sampler began before 2330 hours or after 0030 hours, the sample is INVALID unless the operator can determine that the error in start/stop time was the result of an error in the recorder pen alignment. In such a case the operator should verify the correct start/stop time in the Comments section of the 24-Hour Air Sample Report Form.

2.6 Sample Run Duration

Sample run duration shall be at least 23 hours and no more than 25 hours. Filter samples collected on samplers that operate for less than 23 hours or for more than 25 hours, as documented by the Dickson recorder chart and/or the elapsed time meter, are INVALID.

2.7 Power Failure

If a power failure during a sample run causes the stop time or sample run duration requirements to be violated, the sample is INVALID. Short-term

power outages, however, do not make a sample invalid unless the total time "out" exceeds 1.0 hour.

2.8 Sample Flowrate

If the flowrate through the sampler is outside the accepted range for the site (40.0 CFM ± 10% for PM_{10} and 39 to 60 CFM for TSP, adjusted for altitude if the sampling site is more than 1000 feet above sea-level) for more than one hour during the sampling period, the sample is INVALID. This includes irregular flowrate excursions and the sampler warm-up stabilization period.

2.9 Report Form

The filter is considered INVALID if a completed 24-Hour Air Sample Report Form is not included with the sample.

2.10 Date Sample Received in Lab

A PM_{10} sample is INVALID if not received within 29 days after the sampling date. Mass loss and nitrate loss are shown to be significant after 30 days.

2.11 Filter

A PM_{10} sample is INVALID if a glass filter used in PM_{10} sampling and a TSP sample is INVALID if a quartz filter used in TSP sampling

Appendix B: LAB REMARKS (LIMS) AND COMMENTS (LOGIN) FOR PM_{10} and TSP

This document gives examples of LAB REMARKS to enter when a sample is logged into LIMS, and their related COMMENTS to enter when the sample is logged into the PM_{10} or TSP login Book. The Lab Remarks are in **BOLD** and the login comments are set off by " ". Comments for Invalid samples are entered in the login Book in RED ink; comments for make-up runs are entered in BLUE or BLACK ink.

1. The sampler did not run at all:

   INVALID-SAMPLER DID NOT RUN
   "Sampler did not run" (give detail as to reason, if known)

2. Sampler ran for <23.0 hrs or >25.0 hrs. If the sampler ran for 47.8 hrs:

   INVALID-DURATION OUT OF RANGE
   "Duration out of range; sampler ran 47.8 hrs"

3. The mean flow rate was outside the normal range for the station:

   A. If the flow rate was 30 CFM and too low:

   INVALID-FLOWRATE TOO LOW FOR PM_{10} SAMPLING or
   INVALID-FLOWRATE TOO LOW FOR TSP SAMPLING
   "Flow rate too low @ 30.0 CFM"

   B. If the flow rate was 65.0 CFM and too high:

   INVALID-FLOWRATE TOO HIGH FOR PM_{10} SAMPLING or
   INVALID-FLOWRATE TOO HIGH FOR TSP SAMPLING
   "Flow rate too high @ 65.0 CFM"

4. The filter showed evidence of air leakage due to a faulty gasket:

   INVALID-FILTER SHOWS EVIDENCE OF GASKET LEAKAGE
   "Gasket leakage"

5. No Dickson chart was received with the sample report form and filter, or the Dickson chart received was blank:

   INVALID-CHART RECORD NOT REC'D: F & D NOT VERIFIED

"No Dickson chart" or "Blank Dickson chart"

6. The filter was contaminated before, during or after sampling:

   A. If the filter showed dirt:

      INVALID-FILTER CONTAMINATED W/ DIRT
      "Dirt on filter"

   B. If the filter showed smudges:

      INVALID-FILTER CONTAMINATED W/ SMUDGES
      "Smudges on filter"

   C. If the filter showed fingerprints:

      INVALID-FILTER CONTAMINATED W/ FINGERPRINTERS
      "Fingerprints on filter"

   D. If the filter showed ink stain:

      INVALID-FILTER CONTAMINATED W/ INK STAIN
      "Ink in filter"

   E. If the filter showed other liquid stain:

      INVALID-FILTER CONTAMINATED W/ LIQUID STAIN
      "Stain on filter"

7. The filter was damaged before, during, or after sampling:

   A. If the corner of the filter was torn off:

      INVALID-FILTER DAMAGED: CORNER TORN OFF
      "Corner of filter torn off"

   B. If the edge of the filter was torn:

      INVALID-FILTER DAMAGED: EDGE TORN
      "Edge of filter torn"

   C. If pinhole(s) were in the filter:

INVALID-FILTER DAMAGED: PINHOLE(S)
"Pinhole(s) in filter"

D If the sample area of filter was torn off:

INVALID-FILTER DAMAGED: TORN IN SAMPLE AREA
“Sample area of filter torn”

E If crack was in the sample area of filter:

INVALID-FILTER DAMAGED: CRACK IN SAMPLE AREA
“Crack in the sample area of filter”

8. The flow rate was outside of the normal range for the station for more than one hour (single event or cumulative events). If the flow rate was out of range for 2.5 hrs:

INVALID-FLOWRATE OUT OF RANGE FOR FEW HOURS
"Flow out of range for 2.5 hrs"

9. Sampler started (or ended) more than 30 minutes from midnight (unless the operator noted that the Dickson chart was in error).

A. If the sampler started at 11:15 PM:

INVALID-NON-MIDNIGHT START TIME: >30 MIN
"Start at 2315 hrs"

B. If the sampler stopped at 12:45 AM:

INVALID-NON-MIDNIGHT STOP TIME: >30 MIN
"Stop at 1245 hrs"

10. Sampler ran OK but the Dickson chart pen skipped more than one hour of flow recording (unless the operator noted that the sampler ran OK during the time of the pen skip – see Appendix A, section 2.4 for invalidation criteria based on sampler used). If the Dickson chart pen skipped 2.5 hrs:

INVALID-FLOW NOT RECORDED:CHART PEN SKIP
"Flow not recorded for 2.5 hrs (Dickson chart pen skip)"

11. Sampler was run good, but the center support of the Dickson chart tore out. If the Dickson chart was only able to record 5.5 hours of run time:

    INVALID-FLOW NOT RECORDED: CHART DAMAGED
    "Flow recorded for only 5.5 hrs (Dickson chart damaged)

12. If the glass filter was used in the PM_{10} sampling:

    INVALID-GLASS FILTER USED IN SAMPLING
    “Glass filter used for PM10”

13. If the quartz filter was used in the TSP sampling:

    INVALID-QUARTZ FILTER USED IN SAMPLING
    “Quartz filter used for TSP”

14. If the PM_{10} filter sample was not received by the lab within 29 days:

    INVALID-SAMPLE NOT RECEIVED WITHIN 29 DAYS
    “Sample not received within 29 days”

Appendix C: VISUAL INSPECTION OF QUARTZ MICROFIBER FILTERS USED FOR PM_{10} AND GLASS MICROFIBER FILTER USED FOR TSP SAMPLING

1.0 SCOPE

After receipt from U.S.EPA, all filters are inspected for physical defects before they are sent to sampling sites. This document presents the criteria for the acceptance or rejection of the filters, as adapted from guidelines received from U.S. EPA, Atmospheric Research and Exposure Assessment Lab, QA Support Branch, October 23, 1991.

2.0 PRECAUTIONS

Quartz filters have low tensile strength and are somewhat brittle. They should be handled with great care during inspection, to prevent tearing, breaking, or loss of fibers. To prevent contamination of the quartz filters, plastic gloves MUST be worn when filters are handled. Also, the filters should be protected from any dust or other contaminant until used (i.e., kept in a closed box except during handling).

3.0 INSPECTION METHOD

All filters are inspected using a light screen or table, with critical inspection of BOTH sides of the filter. The following descriptions of visual defects are used to determine the acceptance/rejection of each filter.

3.1 REJECTION CRITERIA:

a) Pinhole - a small hole that can be identified by examining both the front and back of the filter. Any filter containing a pinhole, of any size, is considered a reject.

b) Dense spot - viewed from the filter back, this appears as a dark area (approximately 1/8"-1/4" in diameter) without sharply defined edges. Viewed from the front, an accumulation of filter fibers can be seen. Any filter that contains more than one dense spot is considered a reject.

c) Dark spot - these spots are distinguished from the dense spots in that such dark spots resemble "fly specks". Any filter containing more than two such dark spots is considered a reject.

d) Loose fiber on filter back - this appears as if a rough object had been moved across the filter back and loosened the filter base. If the fibers are large

and/or too numerous to remove without damaging the filter then the filter is considered a reject.

e) Quartz fiber - when viewed from the back this defect resembles a thin spot (see below). The shape can be circular or oval; no evidence of this defect can be seen from the front. When rubbed, the quartz may become detached. If it becomes detached and creates a pinhole, the filter is considered a reject.

f) Coloration - yellow, red, or other colored spots may appear. A filter with such coloration is considered a reject.

g) Other - filter with any obvious structural imperfection not described above, such as frayed edges, torn corners, or indentations or the results of other poor workmanship, is considered a reject.

3.2 ACCEPTABLE IMPERFECTIONS

a) Line - occasionally a fine line created by the manufacturing screen appears across the filter. A filter with such a defect is considered usable.

b) Thin spot - a small area (slightly larger than a pinhole) viewed from the filter back that appears to be weak. More light can be seen through this area than through the surrounding area. Viewed from the front, there is no evidence of this problem. There can be several spots per filter. A filter containing only such defects is considered usable.

d) A filter containing only one dense spot, or two or fewer dark spots is considered usable. A filter that has loose fibers that can be brushed off, or a quartz fiber that can be rubbed off, without damaging the filter is usable.